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METHOD FOR THE PREPARATION OF LOW-TEMPERATURE SINTERED PLANAR
HEXAGONAL IRON-OXYGEN SUBSTANCE BY OXIDE MODIFICATION

Zhang Hongguo, et al.

UNITED STATES PATENT AND TRADEMARK OFFICE
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METHOD FOR THE PREPARATION OF LOW-TEMPERATURE SINTERED PLANAR
HEXAGONAL IRON-OXYGEN SUBSTANCE BY OXIDE MODIFICATION

[Yanghuawu gaixing zhibei di wen shaojie ping mian liujiao tie yang ti de fanghua]

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* * *

Claim

1. A method for the preparation of a low-temperature sintered planar hexagonal iron-oxygen substance by oxide modification, characterized by the fact that said method uses an oxide of Cu or Cu and Zn in the modification of pure Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) to prepare an iron-oxygen substance $(\text{Co}_{2-x-y}\text{Zn}_x\text{Cu}_y)\text{Fe}_{24-\delta}\text{O}_{41}$ powder, with parameters in the formula being $0 \leq x \leq 1$, $0 \leq y \leq 0.8$ and $0 \leq \delta \leq 2$, and its preparation method includes the following steps:

- (1) Appropriate amounts of analytically pure Fe_2O_3 , Co_2O_3 , BaCO_3 , CuO and ZnO are weighed according to stoichiometric ratios;
- (2) Ball milling and mixing are carried out in ethanol by using steel balls as the medium for 12-24 h, followed by stoving and sieving through 60-120 mesh;
- (3) It is heated at a heating rate of $2\text{-}10^\circ\text{C}$ [sic; $2\text{-}10^\circ\text{C}/\text{min}$] to $1000\text{-}1200^\circ\text{C}$ for pre-firing and maintained at that temperature for 2-5 h;
- (4) A flux Bi_2O_3 or V_2O_5 is added at 0-4 wt% to the pre-fired powder from Step (3), followed by ball milling and mixing for 24-72 h, after stoving, it is added to a 6-10 wt% PVA solution at the solution concentration of 4-7%, followed by sieving through 60-120 mesh, granulation and molding; and
- (5) It is heated at a heating rate of $2\text{-}10^\circ\text{C}/\text{min}$ to $800\text{-}950^\circ\text{C}$ and maintained at that temperature for 4-8 h to obtain a planar hexagonal iron-oxygen substance powder.

The present invention relates to a method for the preparation of a low-temperature sintered planar hexagonal iron-oxygen substance by oxide modification. It can be applied appropriately to very high frequency multistage piece-type electrically sensitive parts. It belongs to the field of a new material and a new technology for electronic parts.

With the increasing daily progress in surface-mount technology (SMT), the performance demand with respect to the surface-mount device (SMD) is increased steadily and the cost is decreasing steadily. As one of the important assembly devices of SMDs, piece-type electrical sensitization also has the same development trend. Soft magnetic iron-oxygen substances are the major substrate materials for the manufacture of piece-type electrical sensitization. In order to reduce the cost of parts, at present, only decreasing the iron-oxygen substance sintering temperature and using a relatively low-cost internal electrode material can be selected. The Ag or Ag-Pd alloy electrode is the most commonly used low-cost internal electrode material. However, its melting point does not exceed 961°C . In order to achieve co-sintering of the soft magnetic iron-oxygen substance material and the low melting point internal electrode material and to prevent the diffusion of Ag to the interior inside of the devices, it is necessary to decrease the final sintering temperature of the piece-type electrical sensitization.

The commonly used medium- and high-frequency Ni-Zn-Cu soft magnetic iron-oxygen substances have already passed the preparation of a superfine, high-activity powder, succeeded in achieving low-temperature sintering of piece-type electrical sensitization, and have been used in production. However, in their application to very high frequency, planar hexagonal crystal system Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) type iron-oxygen substances, the low-temperature sintering cannot be achieved to date owing to the complexity in its own structure and composition. With the rapid development in information communication technology, the demand for performance with

respect to the piece-type electrical sensitization has increased steadily. People are eager to realize the low-temperature sintering of the Co_2Z type iron-oxygen substance and market the corresponding multistage piece-type electrical sensitization products.

The sintering temperature for the conventional iron-oxygen substance earthenware and chinaware technology is very high. For Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) type iron-oxygen substance, it can reach 1250-1350°C. In order to lower the earthenware and chinaware sintering temperature, it is often necessary to add a relatively large amount of a glass phase compound or a low-melting-point compound. Although the process is easy to control and understand, some microwave performance, such as quality factors, cutoff frequency, dielectric constant and so on, of the sintered iron-oxygen substance sample are not all ideal. These seriously affect the commercialization of Z-type iron-oxygen substance materials and their parts.

The objective of the present invention is to provide a method for the preparation of a low-temperature sintered planar hexagonal iron-oxygen substance by oxide modification. From the aspect of the structure of the material and on the basis of the conventional iron-oxygen substance earthenware and chinaware sintering technology, the microstructure of the Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) iron-oxygen substance is controlled through the addition of an effective component, so that the melting point of the iron-oxygen substance is lowered after controlling and modification. An appropriate amount of a flux is then added to achieve the objective of lowering the sintering temperature. The sample sintered in this manner has uniform crystal grain dimensions. The density of the material after the low-temperature sintering is also relatively increased. The process is relatively simple. The overall performance of the electrically sensitive parts made is also very good.

The technology designed by the present invention is that an oxide of Cu or Cu and Zn is used in the modification of pure Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) to prepare an iron-oxygen substance ($\text{Co}_{2-x-y}\text{Zn}_x\text{Cu}_y$) $\text{Fe}_{24-\delta}\text{O}_{41}$ powder, with parameters in the formula being $0 \leq x \leq 1$, $0 \leq y \leq 0.8$ and $0 \leq \delta \leq 2$, and its preparation method includes the following steps:

- (1) Appropriate amounts of analytically pure Fe_2O_3 , Co_2O_3 , BaCO_3 , CuO and ZnO are weighed according to stoichiometric ratios;
- (2) Ball milling and mixing are carried out in ethanol by using steel balls as the medium for 12-24 h, followed by stoving and sieving through 60-120 mesh;
- (3) It is heated at a heating rate of 2-10°C to 1000-1200°C for pre-firing and maintained at that temperature for 2-5 h;
- (4) A flux Bi_2O_3 or V_2O_5 is added at 0-4 wt% into the pre-fired powder from Step (3), followed by ball milling and mixing for 24-72 h, after stoving, it is added to a 6-10 wt% PVA solution at the solution concentration of 4-7%, followed by sieving through 60-120 mesh, granulation and molding; and

(5) It is heated at a heating rate of 2-10°C/min to 800-950°C and maintained at that temperature for 4-8 h to obtain a planar hexagonal iron-oxygen substance powder.

The present invention uses an ordinary oxide earthenware and chinaware technological method. It starts with the structure of the material itself without changing the conventional oxide earthenware and chinaware technological flow process. The low-temperature sintering is realized successfully. It enables reduction of the sintering temperature by 400°C or so. Furthermore, the particles after sintering at 900°C or so are relatively small. The crystal size distribution is also relatively uniform. The overall magnetic performance of the material is also better. By using this method, no investment is needed in new production facilities. High-performance multilayer piece-type electrically sensitive parts (MLCI) can be produced without changing the conventional technological method. Since lower-temperature sintering has been realized, a large amount of electrical energy is saved and the facility wear is decreased, thus remarkably decreasing the production costs of the powder and electrically sensitive products.

Explanation of attached figures:

Figure 1 shows the magnetic performance of a sintered iron-oxygen substance sample, in which μ_i represents the initial magnetic conductivity and Q represents a quality factor.

Figure 2 shows the μQ product of the sintered iron-oxygen substance sample.

In the diagrams, the dashed line represents an unmodified sample and a solid line represents a modified sample.

The application examples of the present invention are given in the following.

Application Example 1 ($\text{Ba}_3\text{Co}_{1.8}\text{Cu}_{0.2}\text{Fe}_{23.6}\text{O}_{41}$)

(1) Analytically pure Fe_2O_3 , Co_2O_3 , BaCO_3 and CuO were weighed according to stoichiometric ratios;

(2) Ball milling and mixing were carried out in ethanol by using steel balls as the medium for 24 h, followed by stoving and sieving through 60 mesh;

(3) It was heated at a heating rate of 3°C/min to 1120°C for pre-firing and maintained at that temperature for 3 h;

(4) Bi_2O_3 was added at 2 wt% to the pre-fired powder from Step (3), followed by ball milling and mixing for 48 h, after stoving, it was added to a 6-10% PVA solution at the solution concentration of 5%, followed by sieving through 60 mesh, granulation and molding; and

(5) It was heated at a heating rate of 5°C/min to 880°C and maintained at that temperature in air for 4 h.

For the sintered material, the particles were fine, the distribution was uniform, the density was high, and the overall magnetic performance was excellent. In a very high frequency stage,

the initial magnetic conductivity reached 3.2. The cutoff frequency was increased remarkably, exceeding 1 GHz. The quality factor also showed a pronounced improvement over that of a material not sintered at a low temperature.

Application Example 2 ($\text{Ba}_3\text{Co}_{1.4}\text{Zn}_{0.4}\text{Cu}_{0.2}\text{Fe}_{23.6}\text{O}_{41}$)

(1) Analytically pure Fe_2O_3 , Co_2O_3 , BaCO_3 , CuO and ZnO were weighed according to stoichiometric ratios;

(2) Ball milling and mixing were carried out in ethanol by using steel balls as the medium for 24 h, followed by stoving and sieving through 60 mesh;

(3) It was heated at a heating rate of $2^\circ\text{C}/\text{min}$ to 1140°C for pre-firing and maintained at that temperature for 4 h;

(4) Bi_2O_3 was added at 3 wt% to the pre-fired powder from Step (3), followed by ball milling and mixing for 48 h, after stoving, it was added to a 6-10% PVA solution at the solution concentration of 5%, followed by sieving through 60 mesh, granulation and molding; and

(5) It was heated at a heating rate of $5^\circ\text{C}/\text{min}$ to 875°C and maintained at that temperature in air for 6 h.

For the sintered material, the particles were fine, the distribution was uniform, the density was high, and the overall magnetic performance was excellent. In a very high frequency stage, the initial magnetic conductivity reached 4.0. The cutoff frequency was increased remarkably, exceeding 1 GHz. The quality factor also showed a pronounced improvement over that of a material not sintered at a low temperature.

Application Example 3 ($\text{Ba}_3\text{Co}_{1.2}\text{Zn}_{0.4}\text{Cu}_{0.4}\text{Fe}_{23.6}\text{O}_{41}$):

(1) Analytically pure Fe_2O_3 , Co_2O_3 , BaCO_3 , CuO and ZnO were weighed according to stoichiometric ratios;

(2) Ball milling and mixing were carried out in ethanol by using steel balls as the medium for 24 h, followed by stoving and sieving through 60 mesh;

(3) It was heated at a heating rate of $2^\circ\text{C}/\text{min}$ to 1100°C for pre-firing and maintained at that temperature for 4 h;

(4) V_2O_5 was added at 2 wt% to the pre-fired powder from Step (3), followed by ball milling and mixing for 48 h, after stoving, it was added to a 6-10% PVA solution at the solution concentration of 5%, followed by sieving through 60 mesh, granulation and molding; and

(5) It was heated at a heating rate of $5^\circ\text{C}/\text{min}$ to 875°C and maintained at that temperature in air for 4 h.

For the sintered material, the particles were fine, the distribution was uniform, the density was high, and the overall magnetic performance was excellent. In a very high frequency stage,

the initial magnetic conductivity reached 3.5. The cutoff frequency was increased remarkably, exceeding 1 GHz. The quality factor also showed a pronounced improvement over that of a material not sintered at a low temperature.

Comparative Example 1 ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$):

By using the ordinary iron-oxygen substance sintering technology, appropriate amounts of analytically pure BaCO_3 , Co_2O_3 and Fe_2O_3 were weighed according to molar ratios. Ball milling and mixing were carried out in ethanol by using steel balls as the medium for 24 h. It was pre-fired at 1200°C in air for 3 h. An 8 wt% PVA solution was added to the pre-fired powder at the solution concentration of 5%, followed by sieving through 120 mesh, granulation and molding. It was sintered in air at 1270°C . For the sample, the measured initial magnetic conductivity was 10. The quality factor could not satisfy the usage requirements. The porosity was relatively high. There were even gas pores present inside the crystal grains.

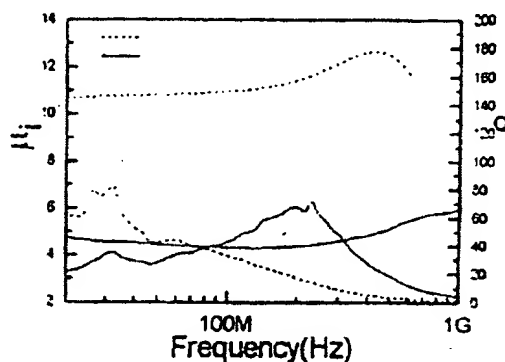


Figure 1

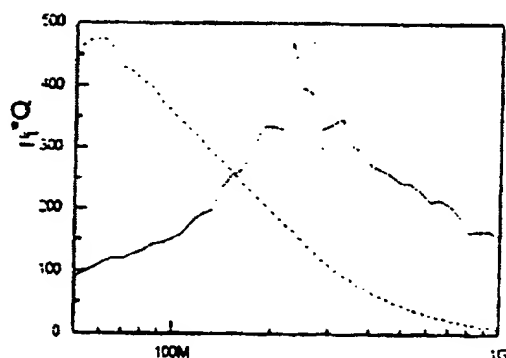


Figure 2

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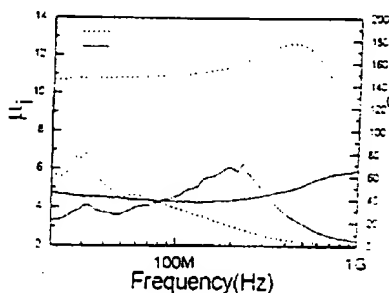
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[54]发明名称 氧化物改性制备低温烧结平面六角铁氧体的方法

[57]摘要

本发明涉及一种氧化物改性制备低温烧结平面六角铁氧体的方法,该方法是首先按化学计量比称取原料,然后在乙醇中以钢珠为媒体球磨,烘干后过筛,然后按一定升温速度升温至 1000—1200℃,保温,最后加入助熔剂,球磨,烘干后再加 PVA 溶液,过筛、造粒、成型再烧结,即制得本发明的产品。本发明实现了低温烧结铁氧体粉,显著降低了粉体及电感产品的生产成本。



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权 利 要 求 书

1、一种氧化物改性制备低温烧结平面六角铁氧体的方法，其特征在于，该方法是用 Cu 或 Cu、Zn 的氧化物对纯 $\text{Co}_2\text{Z}(\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41})$ 改性以制备铁氧体 $(\text{Co}_{2-x-y}\text{Zn}_x\text{Cu}_y)\text{Fe}_{24-3}\text{O}_{41}$ 粉，上式中的各参数分别是 $0 \leq x \leq 1$ ， $0 \leq y \leq 0.8$ ， $0 \leq \delta \leq 2$ ，其制备方法包括下列各步骤：

- (1) 按化学计量比称取适量分析纯的 Fe_2O_3 、 Co_2O_3 、 BaCO_3 、 CuO 、 ZnO ；
- (2) 在乙醇中以钢球为媒体球磨混合 12-24 小时，烘干后过 60-120 目筛；
- (3) 按 $2-10^\circ\text{C}$ 升温速度加热到 $1000-1200^\circ\text{C}$ 预烧保温 2-5 小时；
- (4) 往第 (3) 步预烧粉中加入 0-4wt% 的助熔剂 Bi_2O_3 或 V_2O_5 ，再球磨混合 24-72 小时，烘干后加入 6-10wt% 的 PVA 溶液，溶液的浓度为 4-7%，过 60-120 目筛，造粒，成型；
- (5) 按 $2-10^\circ\text{C}/\text{min}$ 升温速度加热到 $800-950^\circ\text{C}$ ，保温 4-8 小时，即得平面六角铁氧体粉。

说明书

氧化物改性制备低温烧结平面六角铁氧体的方法

本发明涉及一种氧化物改性制备低温烧结平面六角铁氧体的方法，适用于甚高频段的多层片式电感器件，属于电子元器件新材料、新工艺领域。

随着表面安装技术（SMT）的日益发展，对表面安装元件（SMD）的性能要求越来越高，成本却越来越低。作为 SMD 元件的重要组成元件之一，片式电感的发展趋势也是如此。软磁铁氧体是制造片式电感的主要基体材料，为降低元器件的成本，目前只能选择降低铁氧体烧结温度和比较廉价的内电极材料。Ag 或 Ag-Pd 合金电极是最常用的廉价内电极材料，但它的熔点不超过 961℃，为实现软磁铁氧体材料与低熔点内电极材料的共烧结与防止 Ag 向元件内部扩散，必须降低片式电感的最终烧结温度。

常用的中、高频 Ni-Zn-Cu 软磁铁氧体已经通过制备超细高活性粉体成功实现片式电感的低温烧结并应用于生产。但是适用于甚高频段的平面六角晶系 Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) 型软磁铁氧体，由于本身结构与组成的复杂性，一直未能实现低温烧结。随着信息通讯技术的高速发展，对片式电感的性能要求越来越高，人们越来越迫切地渴望实现 Co_2Z 型铁氧体低温烧结并将相应多层片式电感产品投放于市场。

传统的铁氧体陶瓷工艺的烧结温度很高，对于 Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) 铁氧体来说，可达 1250-1350℃，为了降低陶瓷的烧结温度，往往需要添加比较多的玻璃相化合物或低熔点化合物，虽然工艺流程易于控制和掌握，但是所烧结的铁氧体样品的某些微波性能，如品质因数、截止频率、介电常数等均不太理想，严重影响了 Z 型铁氧体材料及其元器件的商品化。

本发明的目的是提出一种氧化物改性制备低温烧结平面六角铁氧体的方法，从材料结构学的角度出发，在传统的铁氧体陶瓷烧结工艺的基础上，通过添加有效组分来调制 Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) 铁氧体的微结构，使调制改性后的铁氧体的熔点降低，然后再适量加入一些助熔剂，从而达到降低烧结温度的目的。这样烧结的样品晶粒尺寸小而均匀，低温烧结后材料的致密度也有了较大的提高，工艺过程相对简单，所做成的电感器件综合性能也很优良。

本发明设计的工艺是用 Cu 或 Cu、Zn 的氧化物对纯 Co_2Z ($\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$) 改性以制备铁氧体 $(\text{Co}_{2-x-y}\text{Zn}_x\text{Cu}_y)\text{Fe}_{24}\text{O}_{41}$ 粉，上式中的各参数分别是 $0 \leq x \leq 1$, $0 \leq y \leq 0.8$, $0 \leq \delta \leq 2$ ，其制备方法包括下列各步骤：

- (1) 按化学计量比称取适量分析纯的 Fe_2O_3 、 Co_2O_3 、 BaCO_3 、 CuO 、 ZnO ；
- (2) 在乙醇中以钢球为媒体球磨混合 12-24 小时，烘干后过 60-120 目筛；
- (3) 按 2-10℃升温速度加热到 1000-1200℃预烧保温 2-5 小时；
- (4) 往第 (3) 步预烧粉中加入 0-4wt% 的助熔剂 Bi_2O_3 或 V_2O_5 ，再球磨混合

24-72 小时，烘干后加入 6-10wt% 的 PVA 溶液，溶液的浓度为 4-7%，过 60-120 目筛，造粒，成型：

(5) 按 2-10℃/min 升温速度加热到 800-950℃，保温 4-8 小时，即得平面六角铁氧体粉。

本发明采用普通的氧化物陶瓷工艺方法，从材料自身结构着手，不改变传统的氧化物陶瓷工艺流程，成功地实现了低温烧结，使烧结温度降低了 400℃ 左右，而且在 900℃ 左右烧结后材料的颗粒比较细小，晶粒分布也比较均匀，材料的综合磁性能也比较优良，采用此法不用投资任何新的生产设备，不用改变传统的工艺方法，即可生产出高性能的多层片式电感元器件 (MLCI)。由于实现了低温烧结，节约了大量电能，减少了设备损耗，从而显著降低了粉体及电感产品的生产成本。

附图说明：

图 1 是烧结铁氧体样品的磁性能，其中 μ_i 表示初始磁导率，Q 表示品质因数。

图 2 是烧结铁氧体样品的 μQ 积。

图中虚线代表非改性样品，实线代表改性样品。

下面介绍本发明的实施例。

实施例 1 ($\text{Ba}_3\text{Co}_{1.5}\text{Cu}_{0.2}\text{Fe}_{23.6}\text{O}_{41}$):

- (1) 按化学计量比称取分析纯的 Fe_2O_3 、 Co_2O_3 、 BaCO_3 、 CuO ；
- (2) 在乙醇中以钢球为媒体球磨混合 24 小时，烘干后过 60 目筛；
- (3) 按 3℃/min 的升温速度加热到 1120℃ 预烧保温 3 小时；
- (4) 往第 (3) 步预烧粉中加入 2wt% 的 Bi_2O_3 ，再球磨混合 48 小时，烘干后加入 6-10% 的 PVA 溶液，溶液浓度为 5%，过 60 目筛，造粒，成型；
- (5) 按 5℃/min 的升温速度加热到 880℃，于空气中保温 4 小时。

烧结后材料的颗粒细、分布均匀，致密度也高，材料的综合磁性能优良，在甚高频段，初始磁导率达到 3.2，截止频率显著提高，超过 1GHz，品质因数也比非低烧材料有明显的改善。

实施例 2 ($\text{Ba}_3\text{Co}_{1.4}\text{Zn}_{0.4}\text{Cu}_{0.2}\text{Fe}_{23.6}\text{O}_{41}$):

- (1) 按化学计量比称取分析纯的 Fe_2O_3 、 Co_2O_3 、 BaCO_3 、 CuO 、 ZnO ；
- (2) 在乙醇中以钢球为媒体球磨混合 24 小时，烘干后过 60 目筛；
- (3) 按 2℃/min 的升温速度加热到 1140℃ 预烧保温 4 小时；
- (4) 往第 (3) 步预烧粉中加入 3wt% 的 Bi_2O_3 ，再球磨混合 48 小时，烘干后加入 6-10% 的 PVA 溶液，溶液浓度为 5%，过 60 目筛，造粒，成型；
- (5) 按 5℃/min 的升温速度加热到 875℃，于空气中保温 6 小时。

烧结后材料的颗粒细、分布均匀，致密度高，材料的综合磁性能优良，在甚高频段，初始磁导率达到 4.0，截止频率显著提高，超过 1GHz，品质因数也比非低烧材料的有明显的改善。

实施例 3 ($\text{Ba}_3\text{Co}_{1.2}\text{Zn}_{0.4}\text{Cu}_{0.4}\text{Fe}_{23.6}\text{O}_{41}$):

- (1) 按化学计量比称取分析纯的 Fe_2O_3 、 Co_2O_3 、 BaCO_3 、 CuO 、 ZnO ；

- (2) 在乙醇中以钢球为媒体球磨混合 24 小时，烘干后过 60 目筛；
- (3) 按 2°C/min 的升温速度加热到 1100°C 预烧保温 4 小时；
- (4) 往第 (3) 步预烧粉中加入 2wt% 的 V_2O_5 ，再球磨混合 48 小时，烘干后加入 6-10% 的 PVA 溶液，溶液浓度为 5%，过 60 目筛，造粒，成型；
- (5) 按 5°C/min 的升温速度加热到 875°C，于空气中保温 4 小时。

烧结后材料的颗粒细、分布均匀，致密度高，材料的综合磁性能优良，在甚高频段，初始磁导率达到 3.5，截止频率显著提高，超过 1GHz，品质因数也比相应非低温烧结材料的有明显的改善。

比较例 1 ($Ba_3Co_2Fe_{24}O_{41}$):

采用普通的铁氧体烧结工艺，按摩尔比称取适量的分析纯原料粉体 $BaCO_3$ 、 Co_2O_3 、 Fe_2O_3 ，在乙醇中以钢球为媒体球磨混合 24 小时，在 1200°C 于空气中预烧 3 小时，往预烧粉中加入 8%wt 的 PVA 溶液，溶液浓度为 5%，过 120 目筛，造粒，成型，于 1270°C 空气中烧结。测得样品的初始磁导率为 10，品质因数则不能满足使用要求，孔隙率比较高，晶粒内部甚至也存在气孔。

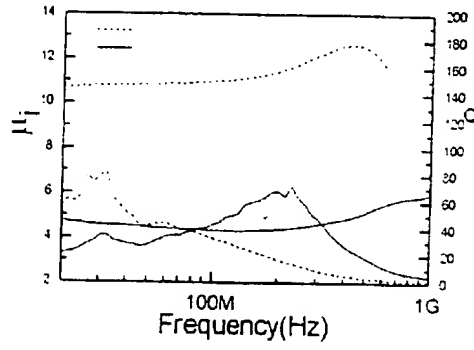


图 1

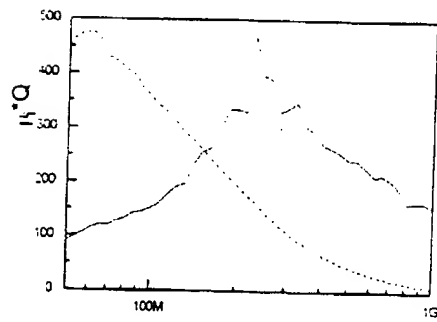


图 2